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ION IMPLANTATION OF CERAMIC BEARINGS

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This report explores the use of ion implantation techniques to form solid lubricating surfaces on all ceramic (silicon nitride) and hybrid ball bearings. Techniques were developed to implant and/or coat silicon nitride and 52100 bearing steel with MoS_2, Boron, and Tin ion beams. A friction/wear tester was also developed to evaluate these coatings in both air and dry nitrogen atmospheres. Friction test results have yielded durable films on both silicon nitride and 52100 steel that exhibit friction coefficients less than 0.1 in a dry state. Oil lubricated controls in a hybrid bearing also show 0.1. Other dry film processes developed have exhibited ultra-low friction (0.02 to 0.05), but these films had limited lifetime. The dry films are fairly durable and adherent, but more work is needed to further improve the durability and adapt the coating process to coating complicated shapes such as balls and rings.

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PREFACE

This final report was prepared by Implant Sciences Corporation, Danvers, MA, under USAF Contract No. F33615-88-C-2857. This contract was initiated under the Small Business Innovation Research (SBIR) Program and sponsored by the Aero Propulsion and Power Laboratory, Wright Research and Development Center, Air Force Systems Command, Wright-Patterson AFB, OH, under the direction of Mr Robert L. Wright (WRDC/POSL) as Project Engineer.

This report covers the Phase I research work conducted during the period of September 1988 to March 1989.

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1.0 PROBLEM SIGNIFICANCE / BACKGROUND

Program Goals

The purpose of the Phase I program was to develop low friction, solid lubricant coatings on ceramic bearing elements. The goal was to use ion implantation to densify and make very adherent coatings. It was a further goal to demonstrate, through laboratory pin-on-disk tests on both all-ceramic and metal-ceramic wear couples, that low friction, high endurance coatings could be produced.

In Phase I three specific processes involving sputter deposition and ion implantation have been shown to provide effective solid lubrication for ceramic bearings. These 3 processes will form the basis for further work on full size bearings during the Phase II program.

2.0 Background

The feasibility of using hot pressed silicon nitride (HPSN) in rolling element bearings has been explored by many researchers over the past 10 years. HPSN offers the opportunity to alleviate many current bearing problems, including DN and fatigue life limitations and extreme environmental demands. The full utilization of these materials in high speed, high temperature applications is partially limited by the availability of suitable lubricants. High coefficients of friction are unacceptable in a truly unlubricated state(1). Ion implantation of lubricious species or ion plating of lubricating compounds are potential approaches to overcome some limitations inherent in conventional lubricating schemes. Bearings for limited life turbomachinery, such as missile propulsion systems and high precision instrument bearings, are restricted by lubrication schemes. High speed missile turbines are being designed without liquid lubrication systems. Conventional dry lubricant powders, MoS_2, intercalated graphites, and elemental metals, are not bonded well and produce debris. Glass bonded lubricants, such as Vitro-lube and Westinghouse Compact, contain materials which may corrode the Si_3N_4 at these temperatures and reduce bearing life. Adherence of the lubricant coatings to the inert ceramic is a major problem that is only partially solved. Transfer films from impregnated cages have been only moderately successful due to limited transfer rate control(2). A second program concluded that significant advances in the separator transfer concept will be required to meet advanced missile engine requirements(3).

Although a variety of materials qualify as solid lubricants in that they exhibit lamellar structure with a low shear strength, getting them to adhere to a substrate in thin film form is not simple. In addition to good thin film adherence, a solid lubricant should have the following 3 properties: low (0.1) to ultra-low (0.01) friction coefficient, good durability, and environmental stability. Solid lubricating films deposited on smooth surfaces, such as bearing balls or raceways, are typically less than 1 micron thick. For such films to be durable under sliding conditions, they must have extraordinarily low wear rates. The actual wear modes of solid lubricating films, like their mechanisms of shear, are not fully understood. Finding one film that ranks high in all three properties is perhaps the main goal of solid lubrication.

Material processing has been the biggest challenge to achieving solid lubrication at high temperatures. High temperatures generally refer to 400^{o}C and above, a range inaccessible to most lubricants that are liquids at room temperature. Materials must not only display low friction coefficients, but also maintain low values over the required temperature range.

This program used low temperature ion implantation / sputtering processes to deposit and/or implant thin films of solid lubricants. The metal member for hybrid bearing applications, typically M50 or 52100 steel, was coated with MoS_2 using ion assisted sputtering. Metal rings were also ion implanted with Tin ions. The silicon nitride elements were ion implanted with Boron or Tin as well as coated with MoS_2.

3.0 PHASE I - R & D PLAN

Our R&D plan was to develop ion implant surface treatments and/or coatings to reduce dry friction in both an all-ceramic bearing and a hybrid bearing. The hybrid chosen for investigation was a bearing with 52100 alloy steel races and silicon nitride balls.

Since our friction measurement method is a ball-on-disk apparatus, all of the surface treatments were on the disk material and not on the ball. The reason for this is that thin coatings must be resident on the moving member of the couple so that the wear is spread over a large area rather than one point on the pin or ball.

A total of 3 surface treatments was selected and developed for each of the 2 types of races. Table 1 below summarizes this.

Race Material	52100	Si_3N_4
1) Boron Ion Implant	-	X
2) Tin Ion Implant	X	X
3) Tin Sputter Coat	X	-
4) MoS_2 Sputter Coat	X	X

The overall plan was to:

1. Develop processes for each of these coatings.

2. Develop a pin-on-disk wear apparatus.

3. Test each of the coatings and measure friction coefficient and coating durability (lifetime).

4. Measure MoS_2 coatings in room air and also in dry nitrogen.

4.0 TEST SPECIMEN PREPARATION

Implant/Coating Development

The coatings developed were tailored to match either the 52100 race or the Si_3N_4 race. For example, the Boron ion implantation was used to form Boron Nitride on the surface of the Si_3N_4 by chemical reaction with the nitrogen. Boron nitride, hexagonal type, is a solid lubricant like graphite. On the other hand, boron ion implantation into 52100 steel was not used because it would merely increase the hardness which is clearly not a solid lubricant.

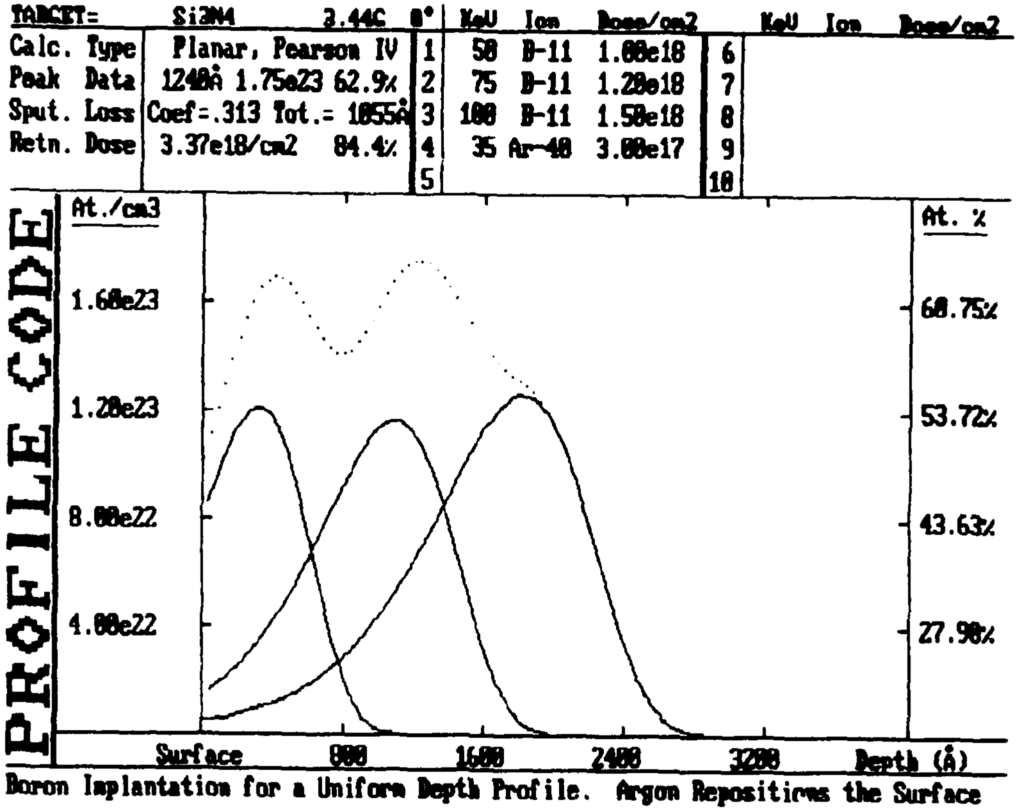

Figure 1. Boron Concentration Vs. Depth for Ion Implantation of 3 Energies in Silicon Nitride. The dotted curve is the sum of the 3 profiles.

Each process was first developed on flat specimens of Si_3N_4 or 52100 to determine useful process parameters and coating deposition rates. Later, actual balls and races from commercial all-ceramic and hybrid bearings were used. All bearings used on the program were donated by Cerbec Ceramic Bearing Co., Windsor, CT, a joint venture of Norton Co. and Torrington Bearing Co.

Boron Ion Implant Process

The Boron ion implantation process was designed to form hexagonal Boron Nitride to a depth of 2400 Angstroms below the surface of the Silicon Nitride. Implant Sciences' PROFILE CODE was used to calculate the 3 boron ion energies which would produce stoichiometric BN with a constant Boron concentration from the surface to the maximum depth available with our ion implantation equipment. Figure 1 shows the calculated implant profiles. The coating obtained was semi-transparent and was very adherent to the Silicon Nitride. As the test data in Section 3.4 will show, the surface was not hexagonal BN but a harder form, and the friction coefficient was only lower by a factor of 2.

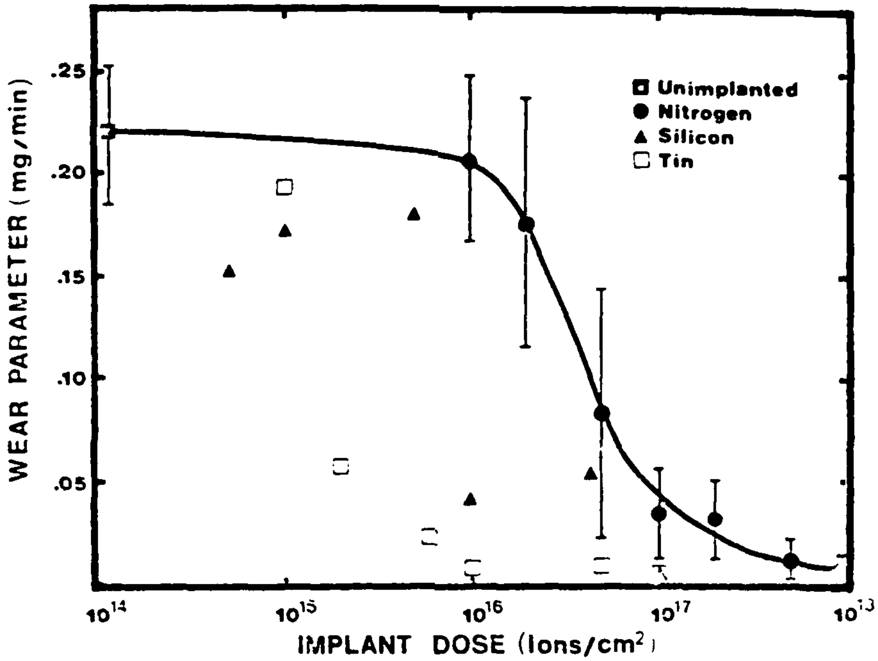

Figure 2. Reduction in Wear By Tin Ion Implantation (Squares) in Steel *As Measured on a Falex Test Machine.* Ref. 4.

Tin (Sn) Ion Implant Process

Tin ion implantation was first used by Baumvol[4] to reduce the friction of steel on steel on a falex wear test machine. The wear versus dose data are shown in Figure 2. Tin oxidizes at low temperatures, and when steel implanted with Tin ions is subject to wear, a thin SnO_2 layer develops at the surface. The oxide is very adherent and continues to diffuse out and grow a new film as the old SnO_2 layer develops at the surfaces. The oxide is very adherent and continues to diffuse out and grow a new film as the old SnO_2 film is worn away. A thin film of SnO_2 is an ideal solid lubricant, a fact well known and used even by Leonardo da Vinci[5].

Ion implantation into both 52100 steel and Si_3N_4 races was done using 200keV Sn^+ ions at a dose of 5E16. This produces a Sn concentration of 10-12 Atomic % extending to a 250 Angstrom depth as shown in Figure 3.

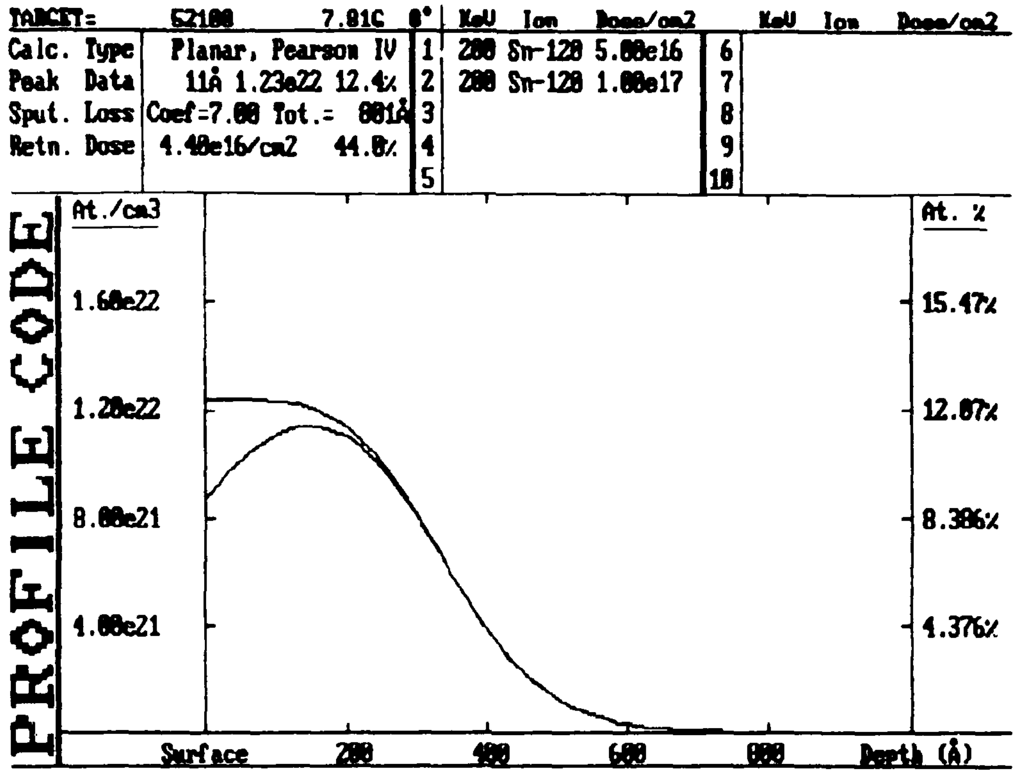

Figure 3. Ion Implanted Profile of Tin Ions Implanted into a 52100 Steel Race at 200 keV Energy. ($5 \times 10^{16}/cm^2$ lower curve, $1 \times 10^{17}/cm^2$ upper curve)

Tin Sputter/Coat/Ion Mix

Since the Tin ion implant saturated at a surface concentration of 12 At%, an attempt was also made to use a Tin coating which would then be driven in with a subsequent ion beam. This process theoretically has the possibility of producing Tin films up to 100% concentration at the surface.

A 500 Angstrom layer of Tin was sputter deposited on a 52100 race and then bombarded with argon at 200keV to a dose of 1E17. This was calculated to drive the Sn coating into the surface and at the same time sputter off the Sn down to the original steel surface. This would have the net effect of producing a Tin implanted profile similar to Figure 3 but having 100% Sn at the surface.

MoS_2 Sputter Coating

We have developed a process which uses dynamical ion mixing (DIM) to both coat a part with MoS_2 and modify its growth characteristics using an ion mix

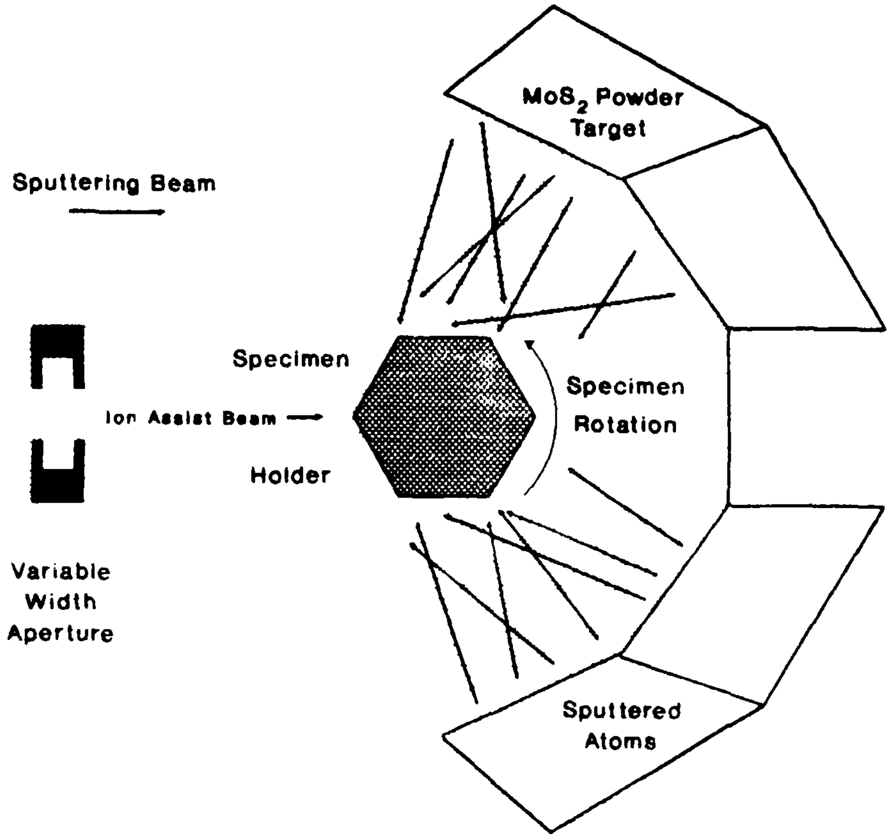

Figure 4. The Ion Assisted Sputter Coating Geometry Used to Deposit the MoS_2 Films on Silicon Nitride and on 52100 Steel.

beam. The process configuration is shown in Figure 4. In this process, a low-cost powder target of MoS_2 can be used. As the beam sweeps from left to right, material is coated on the parts. As the beam sweeps through the center, a small amount of beam impinges on the part to modify the growth properties of the film. This direct beam helps to give the film better adhesion and higher density.

The coating process was used on both the 52100 and the silicon nitride races. After deposition, the races were annealed at 300^{o} in vacuum to completely react all the sulfur. This helps to prevent the film from absorbing moisture from the air.

Figure 5 shows an AES depth profile through the film deposited on a silicon nitride race. The film was approximately 2000 Angstroms thick and shows a stoichiometry of $MoS_{1.6}$. This is typical of a sputtered film. In Phase II, we hope to give these films a supplementary ion implant of 0.4 more sulfur to bring the crystal to MoS_2.

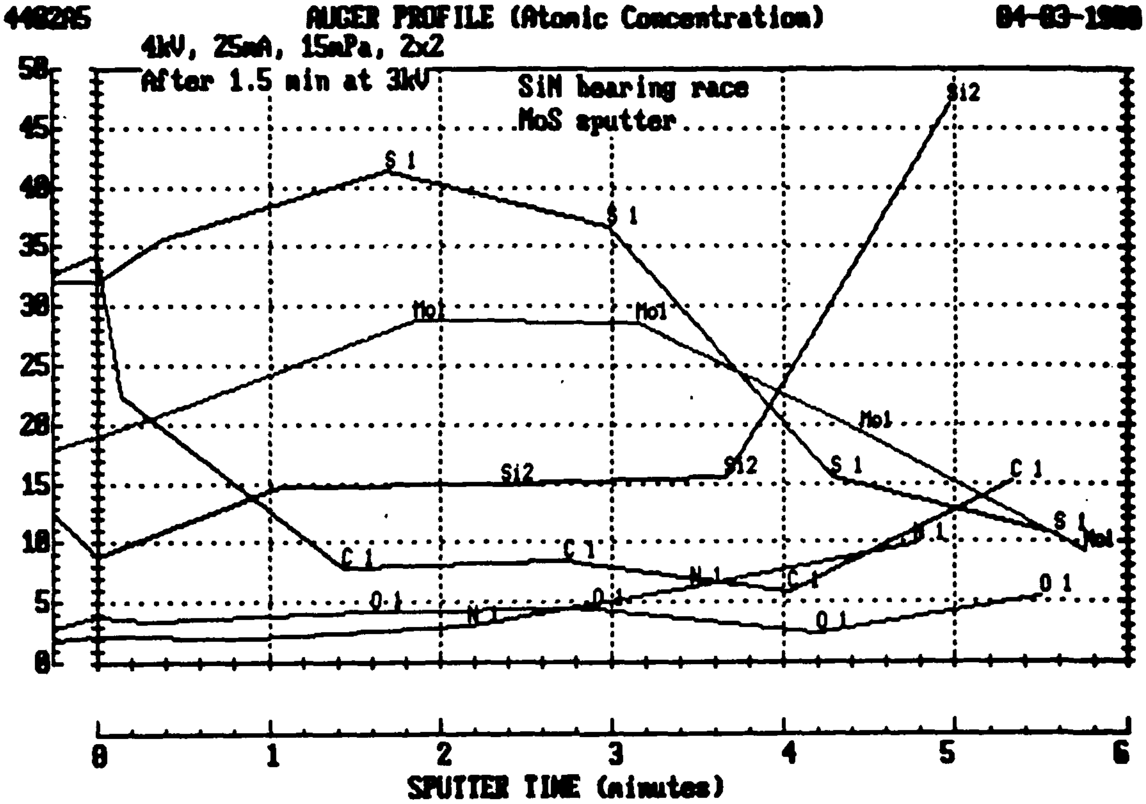

Figure 5. Auger Electron Spectrometry (AES) Depth Profile of MoS_2 Film on a Silicon Nitride Substrate.

5.0 FRICTION TESTER DEVELOPMENT

The system designed and built during this program to obtain the friction data reported here is diagrammed in Figure 6 and photos are shown in Figures 7 to 9. The wear couple consisted of a 52100 steel race against a Grade 5 1/2" Silicon Nitride ball. Tests were also done using a Silicon Nitride race blank against the same ball samples. The balls and both sets of rings were actual bearing components in order to assure the validity of the test results as good indicators of actual in-situ performance.

The rings were inner races (or race blanks) which had been polished on one of their flat, annular surfaces to a finish approximately equivalent to the intended rolling contact region. The rings were sufficiently wide enough to obtain 4 or 5 separate tests on a single sample, utilizing precise incrementation of the turntable. As shown in Figure 8, the balls were mounted in a hexagonal holder held at a 30^{o} angle of contact to the race. This allowed 6 test locations per ball, and with the use of a special jig and the hexagonal flats as position indicators, it was possible

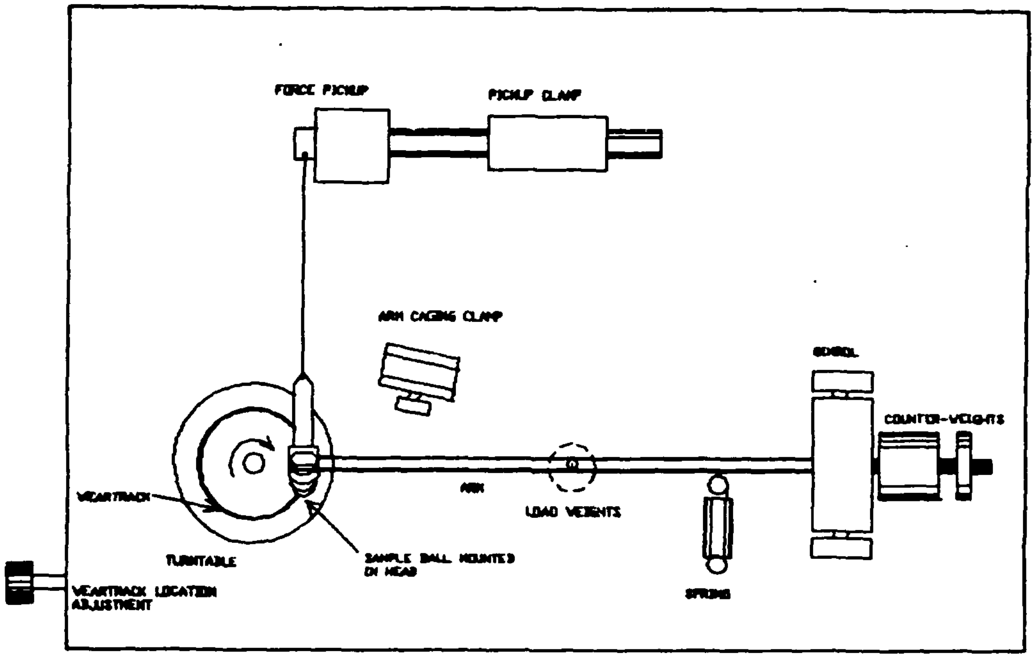

Figure 6. Diagram of Wear/Friction Test Apparatus Constructed for This Program. Top View.

to make accurate microscopic examination and/or ground flat measurement of the contact area for any particular trial.

Friction Test Conditions

In all reported trials, the treatment was applied to the moving race member. Tests were carried out both in air and in a low humidity environment under nitrogen flow. The modified glove box used for this is shown in Figure 9. The bag seen inside the chamber was used to evacuate as much of the internal volume as possible, while purging the box with dry nitrogen simultaneously. This expansion and subsequent deflation process was repeated a total of 5 times, each time removing about 2/3 of the residual internal volume. Thus, we effectively removed almost 95% of the original atmosphere and maintained a slight positive internal pressure of dry N_2 throughout the testing.

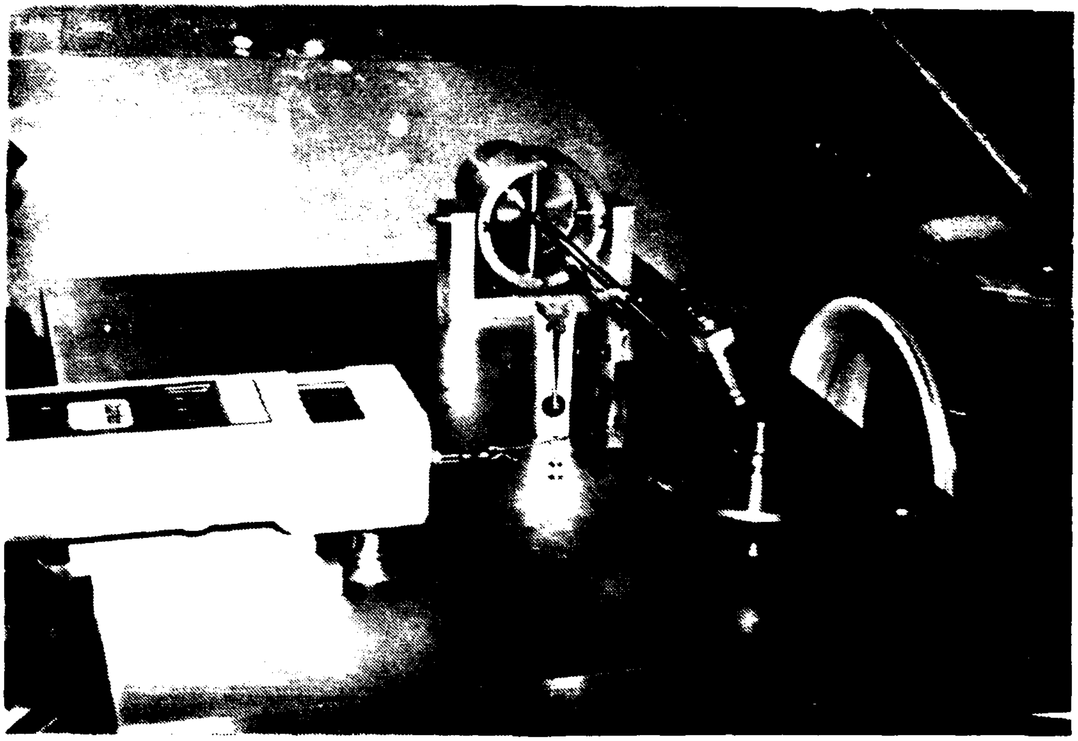

Figure 7. Photograph of Wear/Friction Test Apparatus Showing Friction Force Gauge on the Left.

To maintain constant surface conditions, all samples (ball and ring) were cleaned prior to testing, unless the coating is sensitive to moisture (such as the MoS_2 sputtered films). The cleaning method used was a detergent clean, rinse and nitrogen blow-dry, followed by a TCE vapor degrease and finished with a methanol rinse (and N_2 blow-dry).

The testing conditions used for ceramic-metal couples were a 250 gm load, 7RPM turntable speed, and no lubrication. For a 1/2" diameter Si_3N_4 ball on a 52100 steel plate, the Hertzian stress calculation is shown in Figure 10. The 250g load corresponds to a stress of approximately 0.5GP. For all ceramic couples, the applied load was 100gms to maintain the same Hertzian stress of 0.5Pa. All tests were run for a minimum of 4 minutes, while some of the more successful trials were run for much longer intervals.

The friction force was measured directly by a commercial digital force gauge coupled to an X-Y plotter. The force gauge provided us with accuracy of ± .25%. The gauge was zeroed and the plotter deflection was calibrated before each trial.

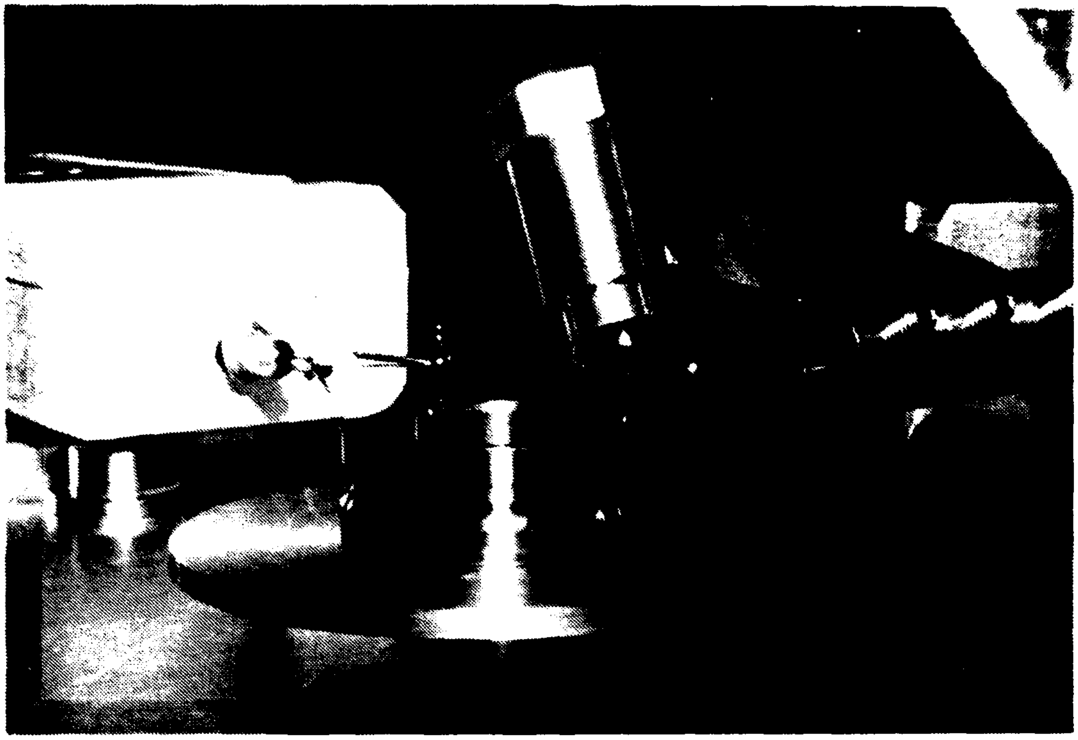

Figure 8. Photograph - Detail of Silicon Nitride Ball on 52100 Race (Disk).

The latter was accomplished by hanging a known weight from the unit being held vertically and adjusting the plotter vernier to give the exact force measurement.

6.0 TEST RESULTS

Friction data was taken on each coating while the race was moving at 7RPM relative to the stationary ball. This corresponded to a linear sliding speed of 1.25cm/sec on the 52100 race and 1.0cm/sec on the Si_3N_4 race. The hertz stress of a ball on a plate was calculated to be 0.43GPa for the 52100 race and 0.47GPa for the Si_3N_4 race. All tests were done dry, without lubricant on the coatings. Only the control (No coating) on the 52100 race was run with oil lubricant to serve as a low friction goal for the dry lubricants. The data show that several of our dry film lubricants had lower friction than the oil lubricated control.

The results for the 52100 race against a Si_3N_4 ball are summarized in Table 2. The No coating (dry) control quickly developed a 0.4 friction coefficient which is clearly not the mode in which a hybrid bearing is designed to operate. The oil

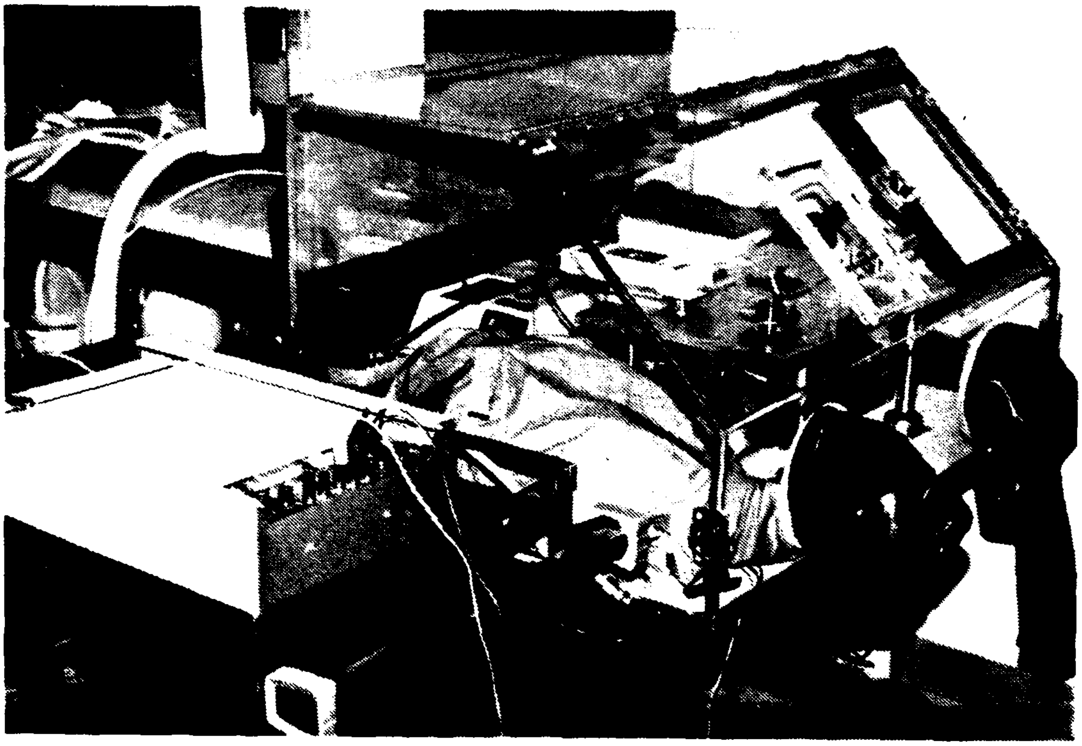

Figure 9. Photograph of Wear/Friction Test Apparatus in Environmental Control Glove Box.

lubricated control maintained a steady 0.1 coefficient as long as the oil was continuously supplied.

The MoS_2 sputter coating in dry N_2, after a short break-in period, achieved a friction coefficient of 0.05, which is 1/2 that of an oil lubricant. Moreover, the solid lubricant film did not break down and continued to maintain this low value for several hours until we ran out of plotter paper. In air, however, the MoS_2 coating is attacked by moisture which modifies the lubrication properties. In air, the MoS_2 is comparable to the oil lubricant and much better than the dry uncoated data. The chart recorder data for these 4 runs is shown in Figures 11 through 14.

The results for the all-ceramic case (Si_3N_4 race against a Si_3N_4 ball) are summarized in Table 3. In this case, none of the controls was oil lubricated. The dry control (uncoated) quickly came to a friction coefficient of 0.35. The boron implanted race showed about half the friction coefficient of the control. The low dose tin showed an extremely low initial value of 0.02, but the coating quickly

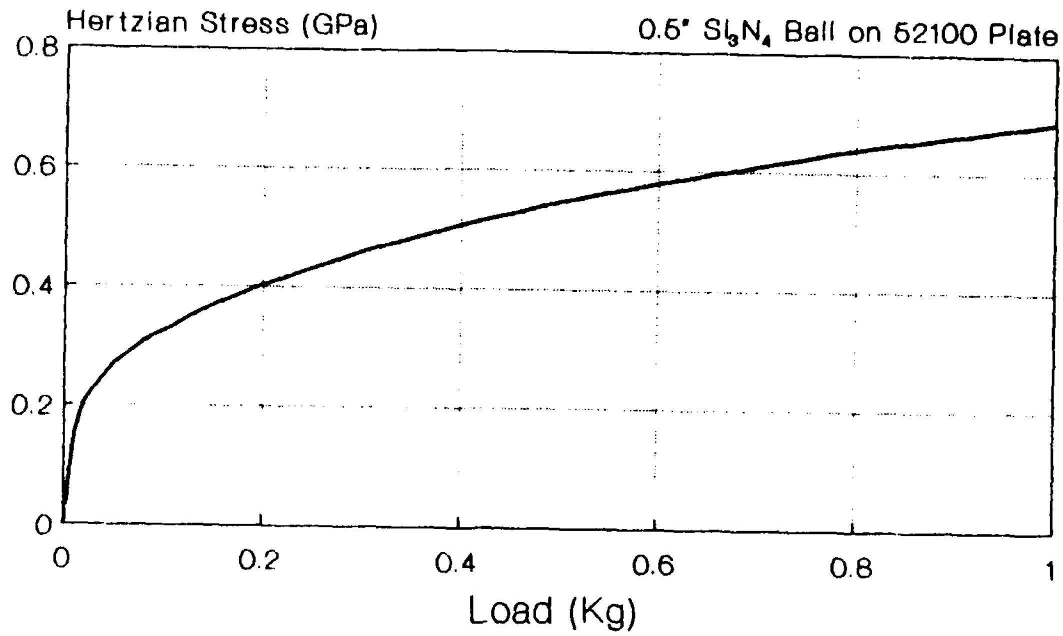

Figure 10. Calculation of Hertzian Stress for a 0.5" Diameter Silicon Nitride Ball on a 52100 Steel Plate.

broke down in 1.25 minutes and reverted to the uncoated case. The higher dose Sn implant did not show this extremely low friction. This Sn implant requires some more development to realize both the low friction and long life simultaneously.

Both of the MoS_2 coatings on the Si_3N_4 races showed greatly reduced friction and no breakdown of the coating. The test in dry N_2, however, did give lower values (0.1), which is consistent with the theory that the MoS_2 film is attacked by moisture. The chart recorder data for these runs are shown in Figures 15 through 20.

Photomicrographs of selected wear tracks are shown in Figures 21 through 24. Figure 21 shows the track in an unlubricated control of 52100. Note that with lubricant, the silicon nitride ball tears up the steel surface. Figure 22 shows the tin ion implanted silicon nitride disk. Figure 23 shows the MoS_2 coating on the

FRICTION TEST DATA
(52100 Race on Si_3N_4 Ball)

Coating	Speed	Hertz Stress (Peak)	Friction Coefficient	Breakdown Time	Figure
No Coating (Dry)	1.25 cm/sec	0.43GPa	0.2 → 0.4	—	10
No Coating (Oil)	1.25 cm/sec	0.43GPa	0.1	—	11
MoS_2 (Air)	1.25 cm/sec	0.43GPa	0.1 → 0.2	None	12
MoS_2 (N_2)	1.25 cm/sec	0.43GPa	0.2 → 0.05	None	13

Table 2. Data Summary for 52100 Disk and Silicon Nitride Pin

52100 disk in both dry Nitrogen (Top in photo) and room air (Bottom in photo). Note that the integrity of the track is preserved much better in the dry nitrogen (no humidity) case. Figure 24 shows a similar set of tracks for the silicon nitride disk. In Air the MoS_2 film is not intact.

7.0 CONCLUSIONS AND RECOMMENDATIONS

The program has accomplished some fairly significant advances in the science of dry ceramic tribology. Some promising basic dry lubricant coatings, and the tools, apparatus, and techniques to evaluate them in the laboratory, have been developed. These tools and techniques have also been developed with the expert advice and technical input from the leading ceramic bearing manufacturer, Cerbec.

Specifically, the major accomplishments of the Phase I effort were:

1. Design and fabrication of a friction/wear pin-on-disk tester which was tailored to test ceramic ball bearing components. The tester also can

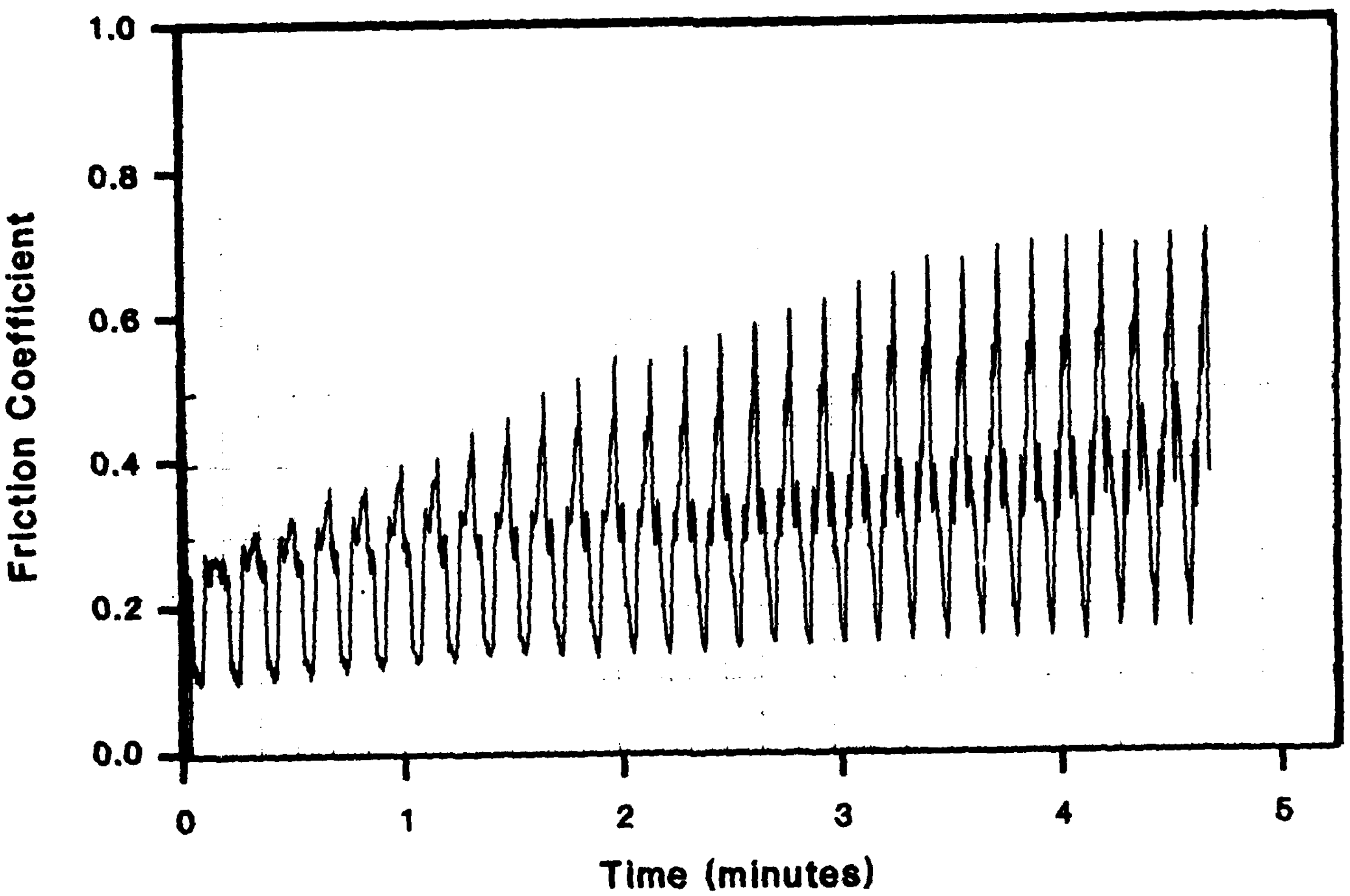

Figure 11. Friction Measurement, Silicon Nitride Ball on 52100 Disk, 7RPM Unlubricated, Uncoated Control.

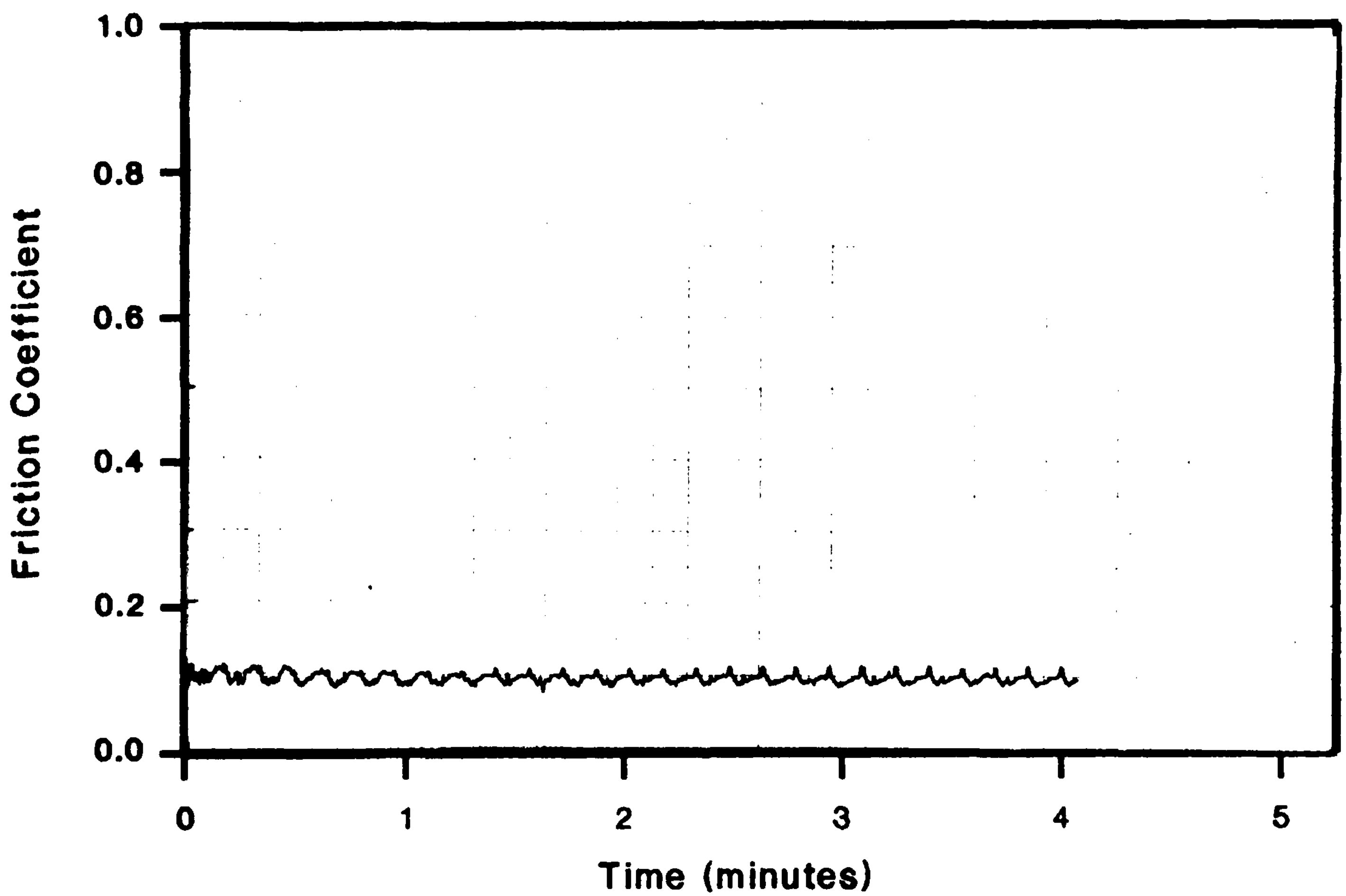

Figure 12. Friction Measurement, Silicon Nitride Ball on 52100 Disk, 7RPM Oil Lubricated, Uncoated Control.

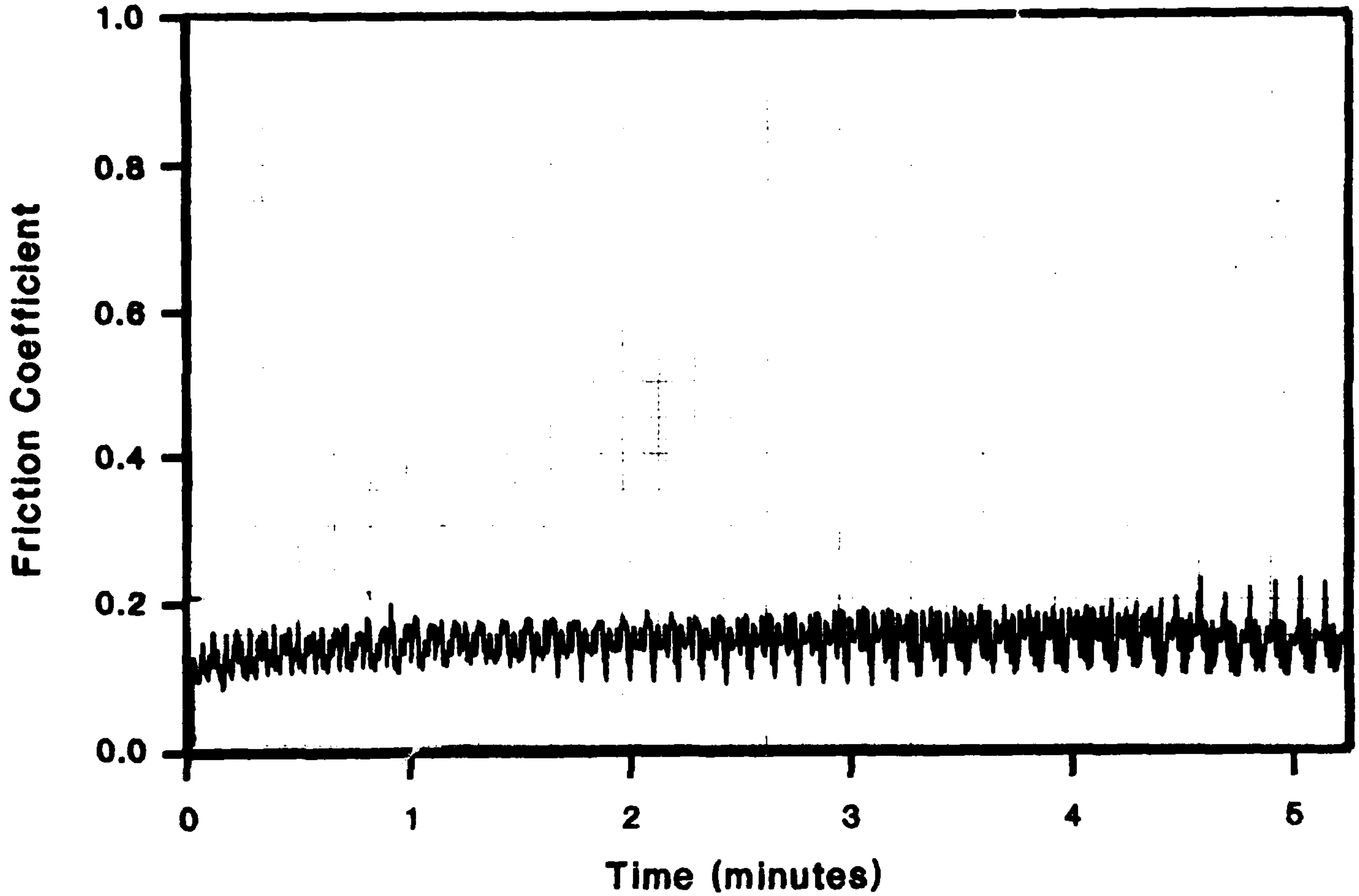

Figure 13. Friction Measurement, Silicon Nitride Ball on 52100 Disk, 7RPM MoS_2 Coated, in Air.

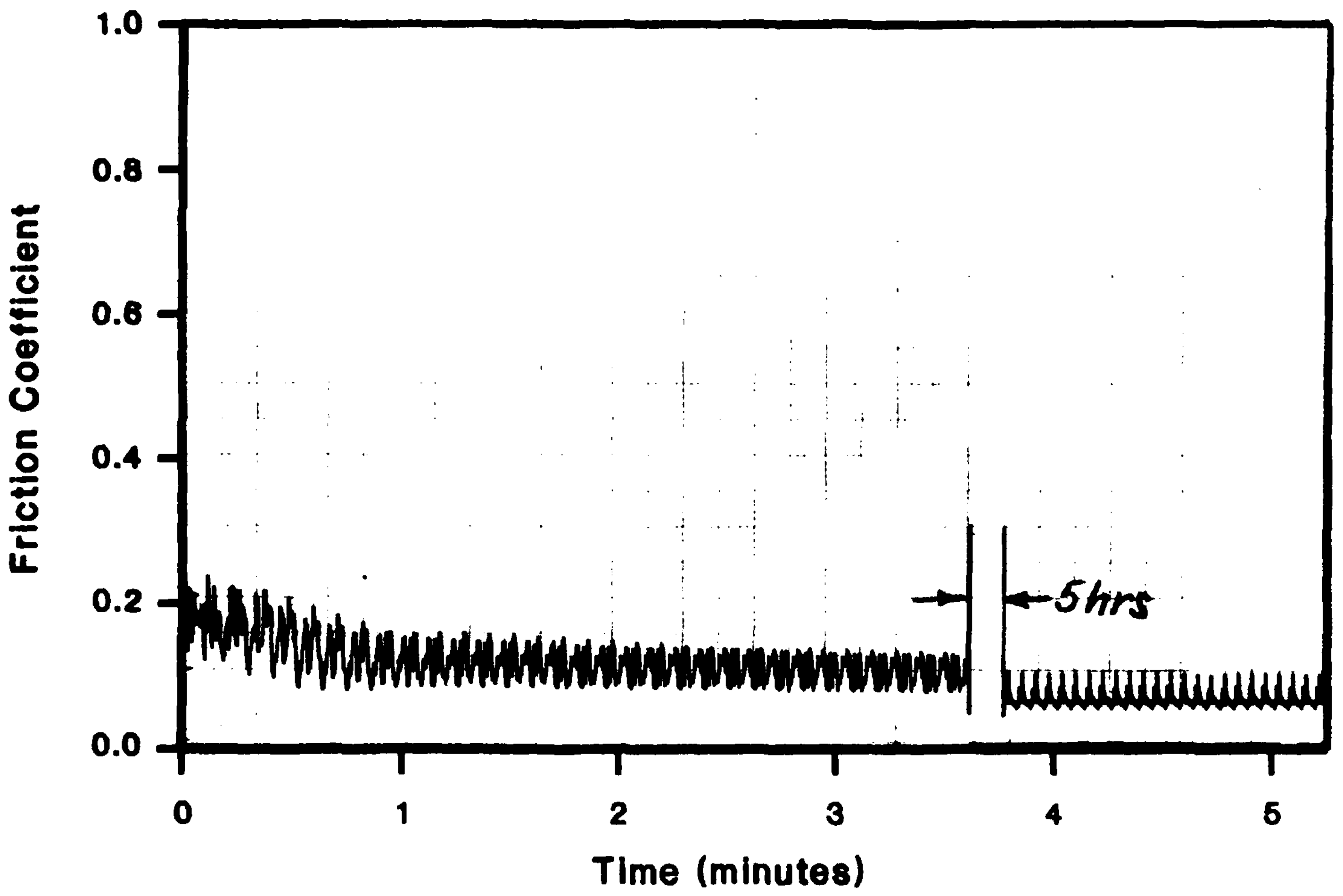

Figure 14. Friction Measurement, Silicon Nitride Ball on 52100 Disk, 7RPM MoS_2 Coated, in Dry N_2.

FRICTION TEST DATA
(Si_3N_4 Race on Si_3N_4 Ball)

Coating	Speed	Hertz Stress (Peak)	Friction Coefficient	Breakdown Time	Figure
No Coating (Dry)	1.00 cm/sec	0.47GPa	0.2 ⟶ 0.35	—	14
Boron Implanted	1.00 cm/sec	0.47GPa	0.12 ⟶ 0.15	Not Tested	15
Tin Implanted (Low Dose)	1.00 cm/sec	0.47GPa	0.02 ⟶ 0.05	1.25 min	16
Tin Implanted (High Dose)	1.00 cm/sec	0.47GPa	0.1 ⟶ 0.3	7 min	17
MoS_2 (Air)	1.00 cm/sec	0.47GPa	0.1 ⟶ 0.15	None	18
MoS_2 (N_2)	1.00 cm/sec	0.47GPa	0.1 ⟶ 0.1	None	19

Table 3. Data Summary for Silicon Nitride Pin and Disk

provide data in various environmental conditions such as humid air, dry N_2, Argon, and high vacuum.

2. A large library of baseline friction/wear data has been generated so that the efficacy of any solid lubricants can be compared to baseline.

3. The program has developed 3 solid lubricant coatings that have shown promising friction reduction and should be pursued further. These are boron ion implant and Tin ion implant on Si_3N_4 and MoS_2 dynamically ion mixed coatings on both Si_3N_4 and bearing steel substrates.

Dry friction coefficients much lower than oil lubricated controls have been observed.

These accomplishments warrant further resources to develop these surface coatings into a viable ceramic bearing system that can eventually be used in higher temperature Air Force engines of all kinds.

A Phase II program should be initiated which will address the following:

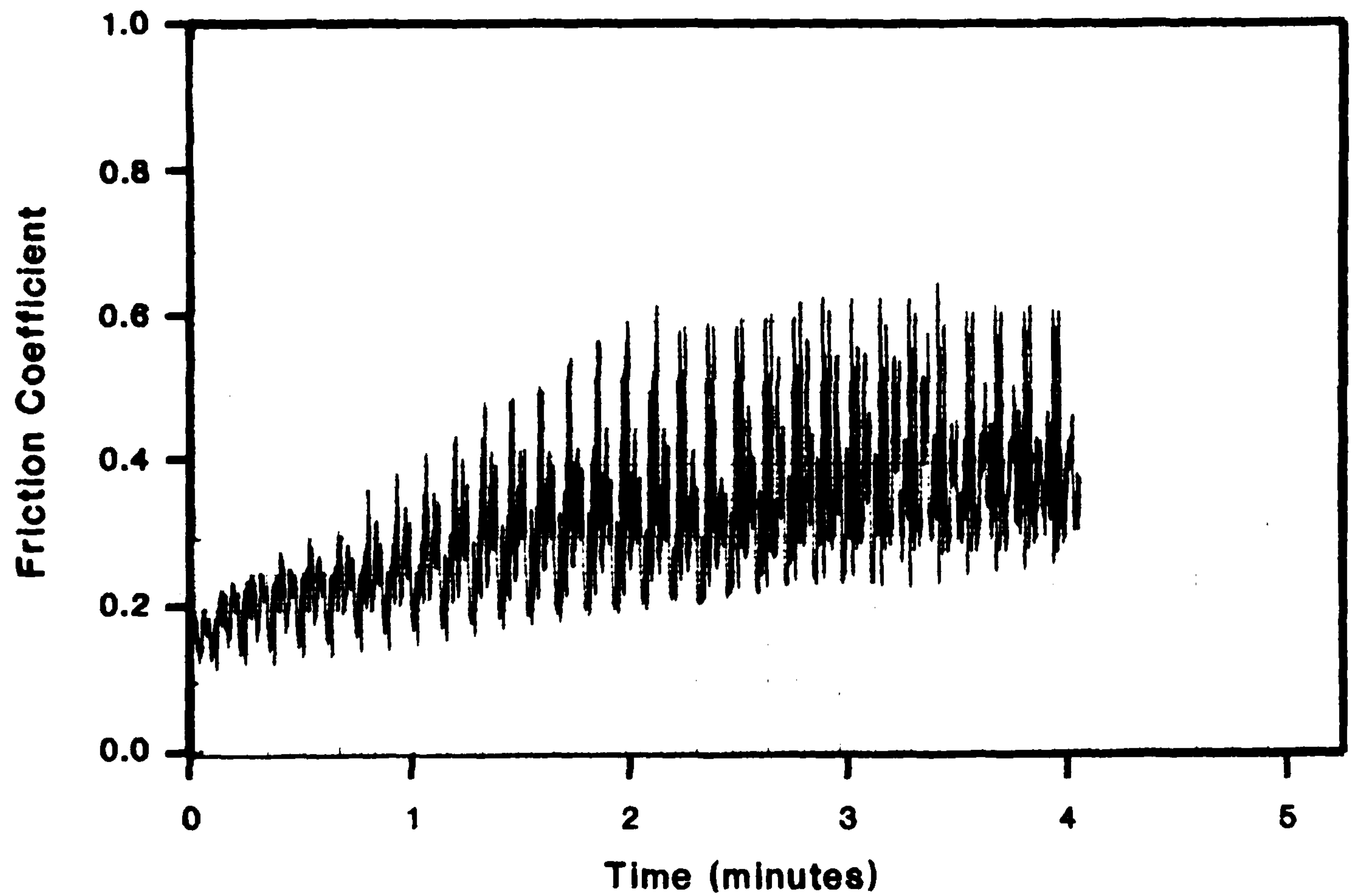

Figure 15. Friction Measurement, Silicon Nitride Ball on Silicon Nitride Disk, 7RPM Uniubricated, Uncoated Control.

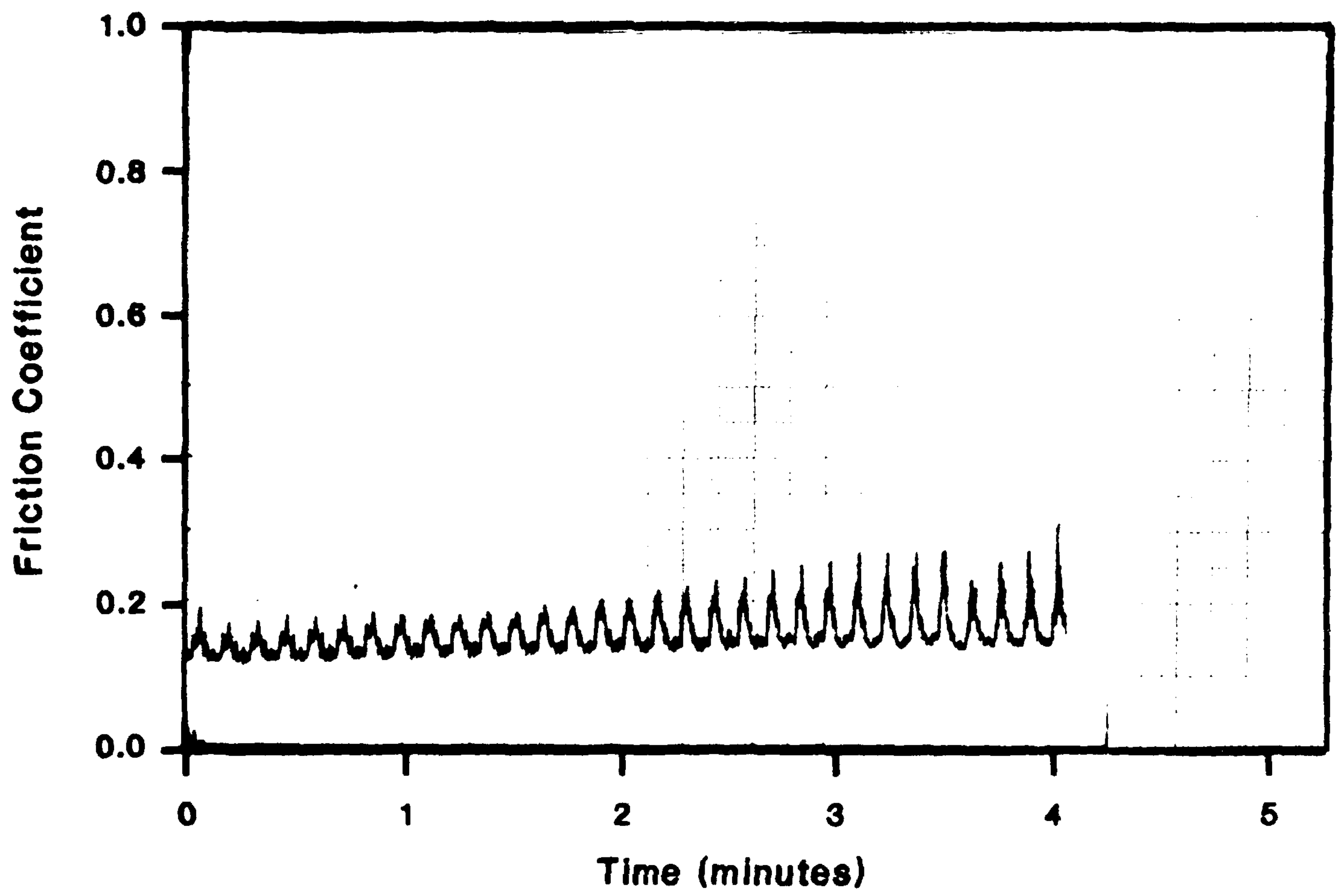

Figure 16. Friction Measurement, Silicon Nitride Ball on Boron Implanted Silicon Nitride Disk, 7RPM, Uncoated.

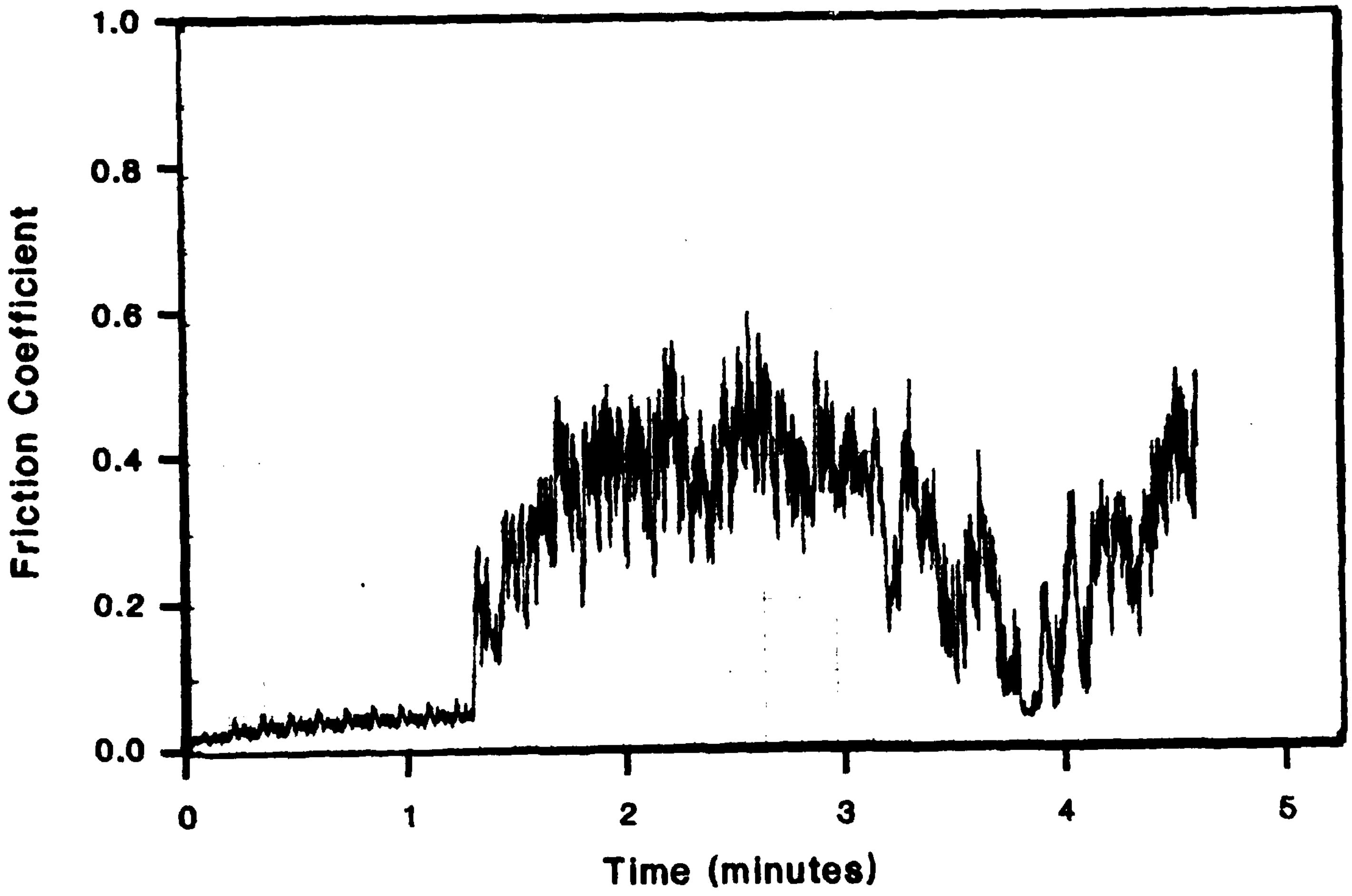

Figure 17. Friction Measurement, Silicon Nitride Ball on Tin Ion Implanted Silicon Nitride Disk, 7RPM, Dose of 5×10^{16} Ions/cm^2.

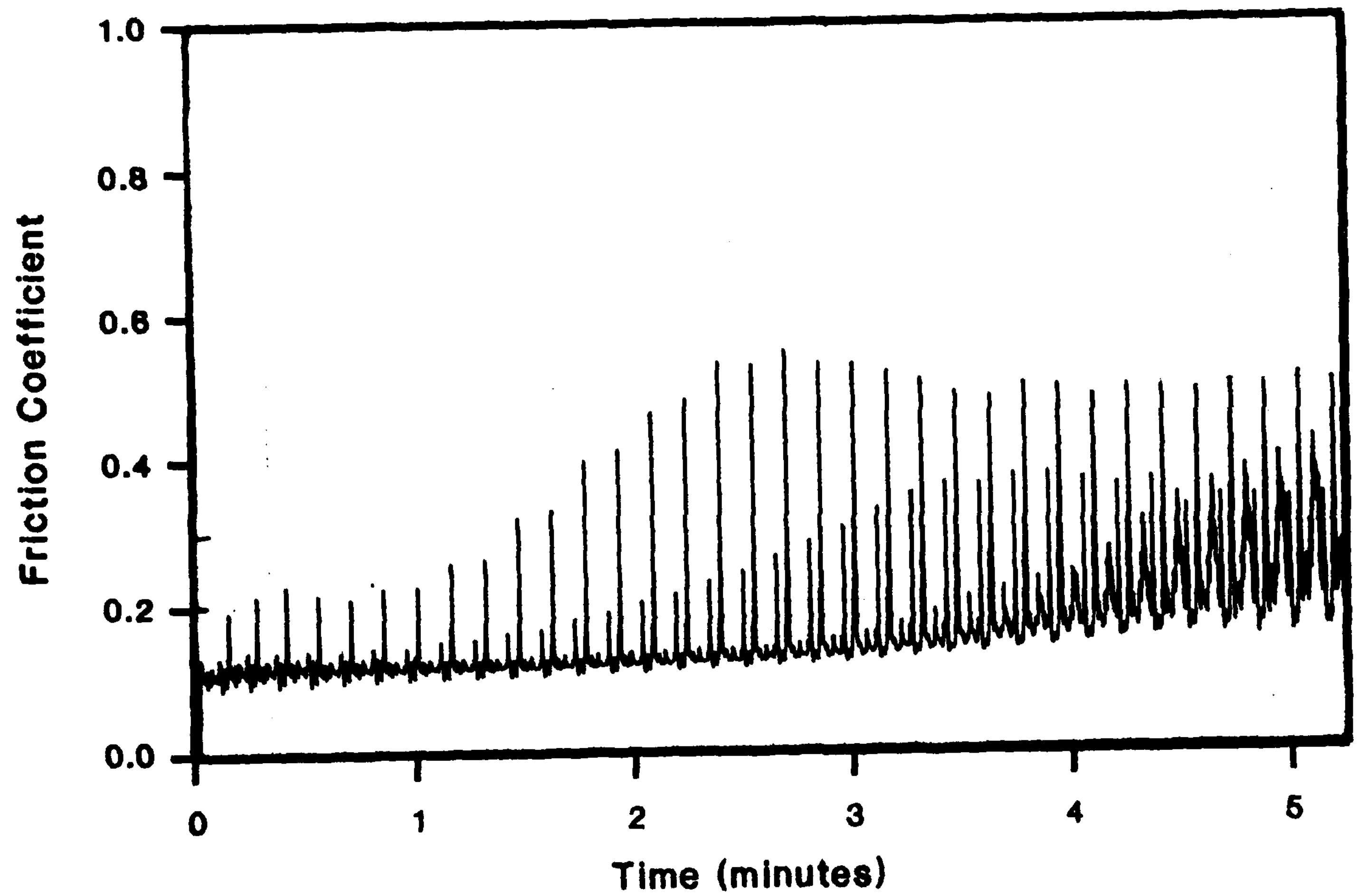

Figure 18. Friction Measurement, Silicon Nitride Ball on Tin Ion Implanted Silicon Nitride Disk, 7RPM, Dose of 1 x 10^{17} Ions/cm^2.

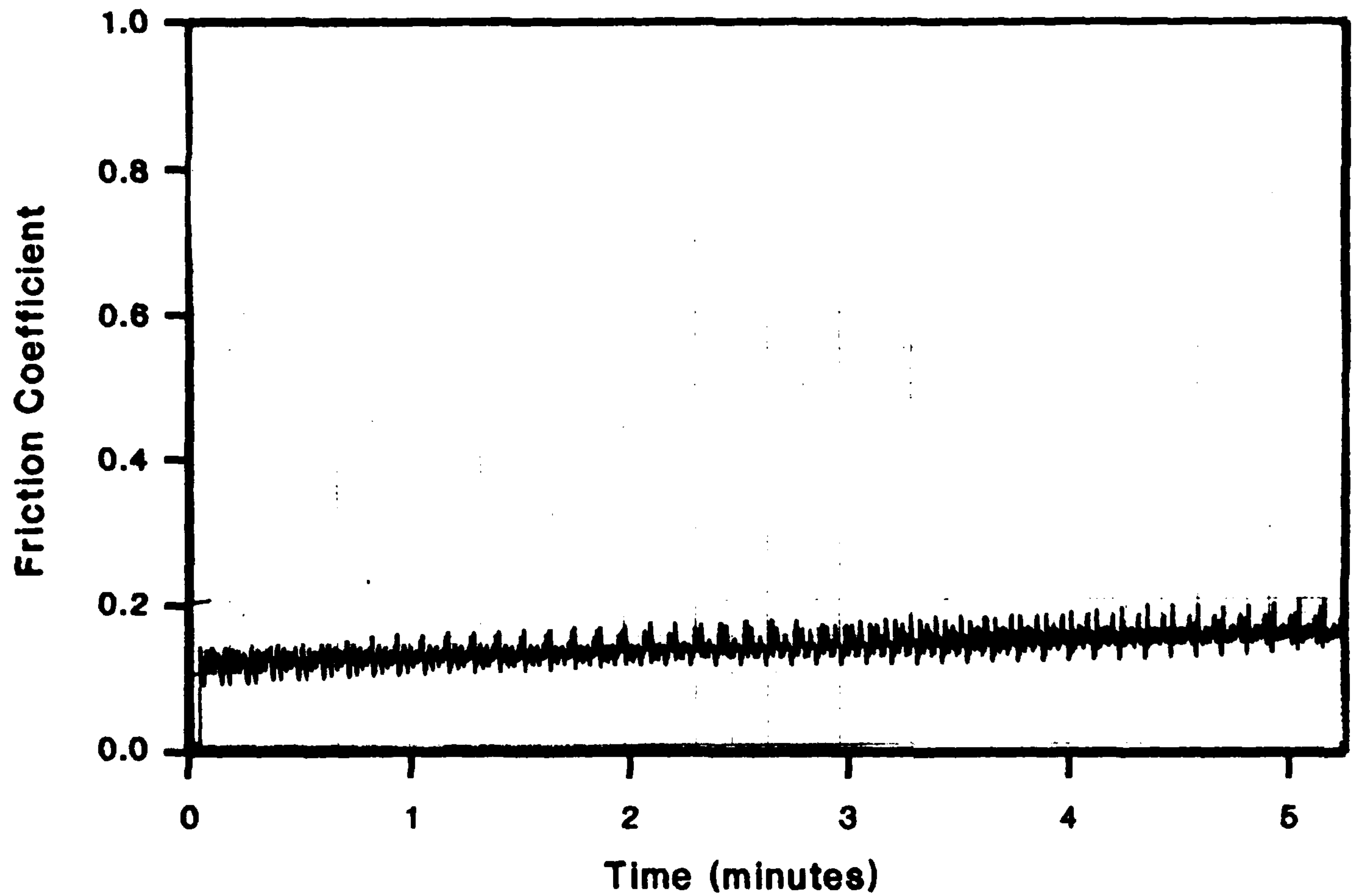

Figure 19. Friction Measurement, Silicon Nitride Ball on Silicon Nitride Disk, 7RPM, MoS_2 Coated, in Air.

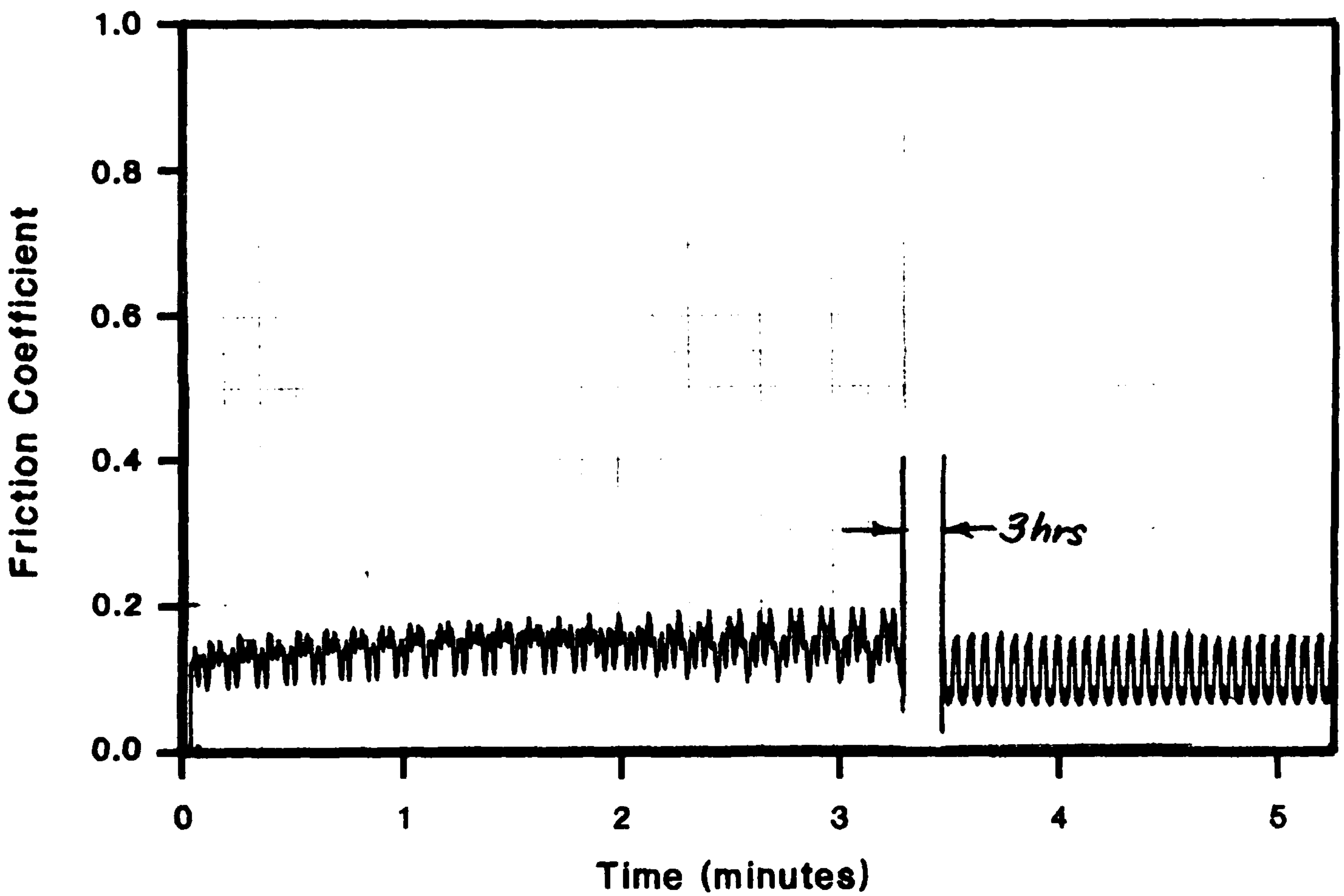

Figure 20. Friction Measurement, Silicon Nitride Ball on Silicon Nitride Disk, 7RPM MoS_2 Coated, in Dry N_2. Note the break in the time scale showing extremely low friction without film breakdown.

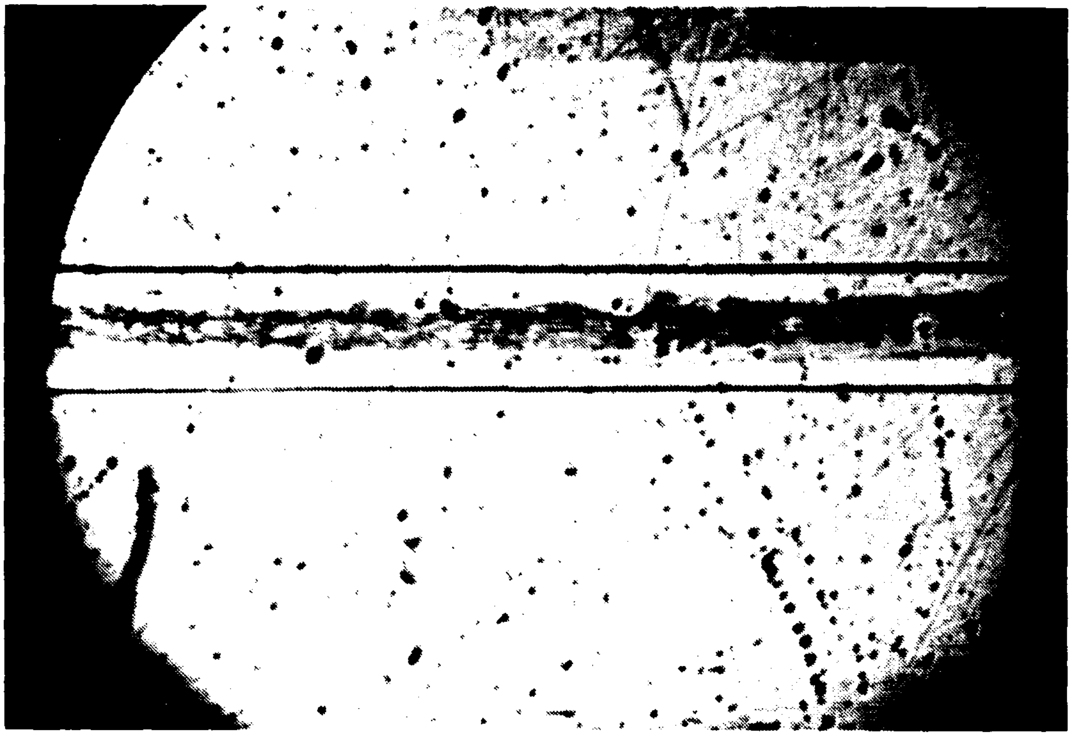

Figure 21. Wear Groove of 52100 Steel Disk - Dry Control

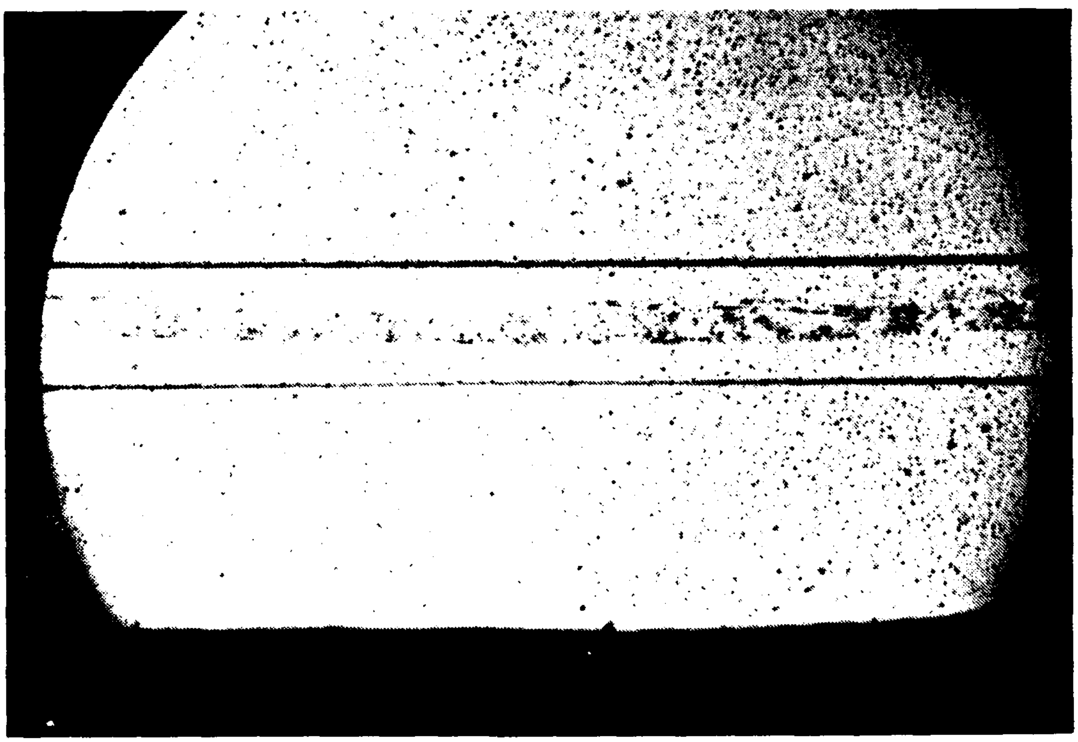

Figure 22. Wear Groove of 52100 Steel Disk - Tin Implanted at 5 x $10^{16}/cm^2$ Dose.

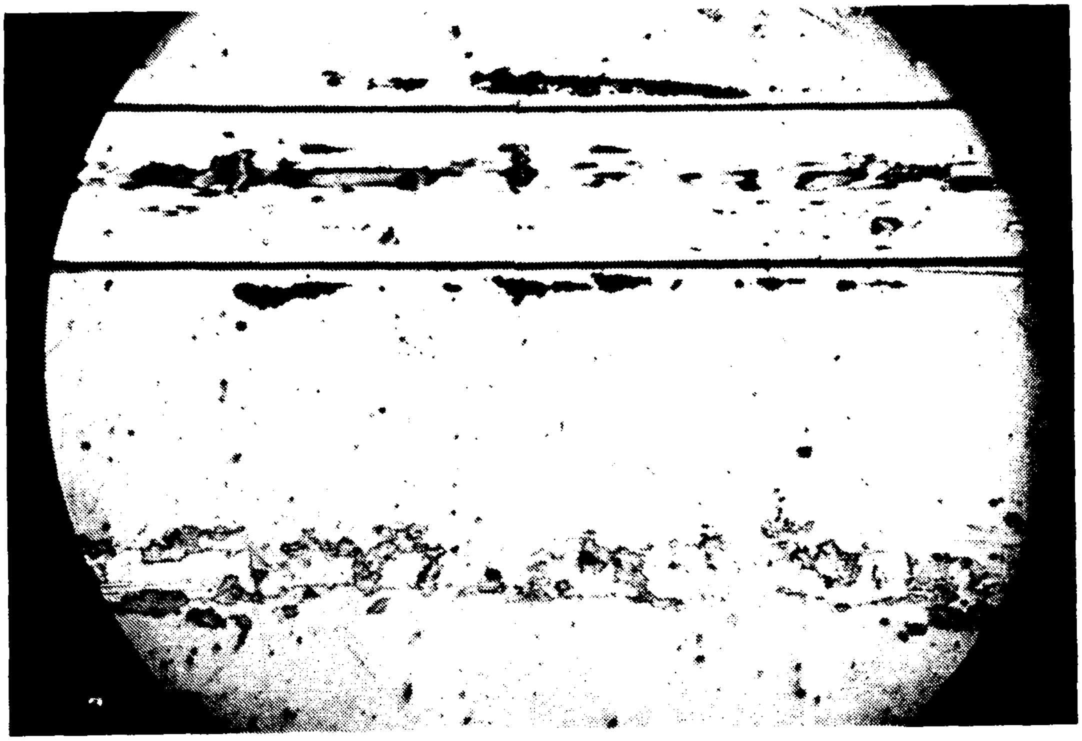

Figure 23. Wear Groove in 52100 Steel Disk. Top track is Silicon Nitride ball sliding in dry Nitrogen atmosphere. Bottom track is Silicon Nitride ball sliding in air.

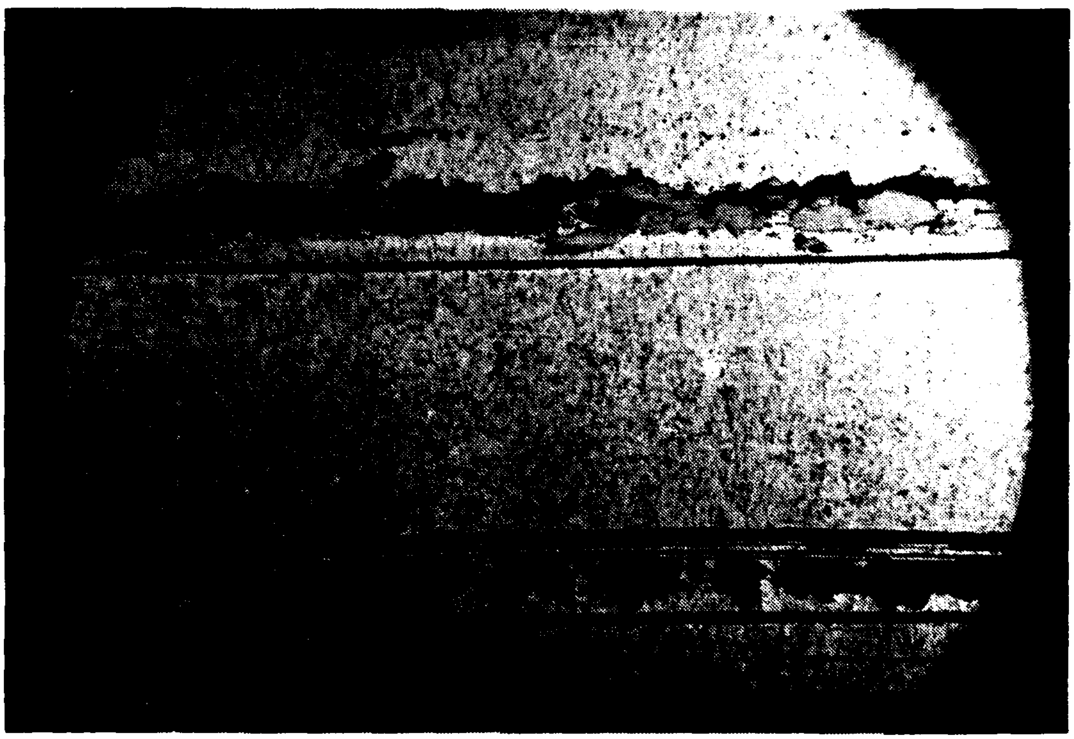

Figure 24. Wear Groove in Silicon Nitride Disk. Top trac is Silicon Nitride ball sliding in dry nitrogen atmosphere. Bottom track is Silicon Nitride ball sliding in air.

1. Improvement of the dynamic ion mixing process to develop better morphology, more adherent, higher stoichiometry MoS_2 films. Also, some work should be done with additives or covers on the MoS_2 to protect the film from moisture attack.

2. Further development of the Tin ion implant process parameters to achieve a more durable surface so that the amazingly low dry friction coefficient observed (<0.02) can be maintained for a longer time. The tin ion implant process has great potential because this coating "grows" new solid lubricant by diffusion as the coating is worn away.

3. After the basic coatings/ion implant processes are developed, combinations of treatments should be tested on the pin-on-disk apparatus. Particularly interesting is a boron implant on the Si_3N_4 ball and a MoS_2 coating on a steel race. This combination has the possibility of accelerating the introduction of a hybrid bearing into the engine community.

4. One necessary requirement for introduction of any dry lubricant treatment is to demonstrate the benefit on real components, operating in realistic conditions. In order to do this, the processes must be adapted to coat races and balls efficiently and then a full scale bearing must be run to gather operating data. In Phase II, we plan to develop apparatus and fixtures to coat full scale bearings, and in conjunction with Cerbec, run some full scale bearing tests.

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